

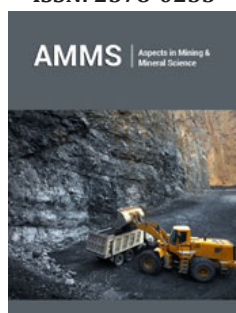
SEM-EDS Microanalysis of Sedimentary and Felsic Volcanic Units in Uruguay: A New Analytical Approach for Pozzolan Resource Assessment

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ISSN: 2578-0255



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Submission:  May 11, 2026

Published:  May 22, 2026

Volume 15 - Issue 1

How to cite this article: Gonzalo Blanco*, Paulina Abre, Camila Zunino and Sebastián Mármol. SEM-EDS Microanalysis of Sedimentary and Felsic Volcanic Units in Uruguay: A New Analytical Approach for Pozzolan Resource Assessment. *Aspects Min Miner Sci.* 15(1). AMMS. 000853. 2026.

DOI: [10.31031/AMMS.2026.15.000853](https://doi.org/10.31031/AMMS.2026.15.000853)

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Abstract

The mineralogy and major element distribution of selected samples from 13 localities, comprising sedimentary and felsic volcanic rocks, are evaluated using Scanning Electron Microscopy Equipped with An Energy-Dispersive X-ray Spectrometry (SEM-EDS). This study aimed to characterize clay textures, identify mineral phases, and determine elemental composition and distribution. Precambrian pelites and tuffs show a composition dominated by illite, quartz and feldspar. Paleozoic sedimentary rocks show variations in clay composition: the Cordobés Formation is predominantly composed of kaolinite, while the Yaguarí Formation is characterized by montmorillonite. Rhyolites from the Lower Cretaceous Arequita Formation are composed of quartz, sanidine, volcanic glass and alteration product such as kaolinite and illite-smectite. The summatory of $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ above 70%, combined with the fineness and mineralogy of all analyzed rocks, when compared to similar examples from the literature, suggests their potential use as natural pozzolans after thermal treatment.

Keywords: SEM-EDS; Mineralogical characterization; Natural pozzolans; Felsic volcanic rocks; Sedimentary rocks; Uruguay

Introduction

The characterization and use of natural pozzolans as aggregate in Portland cement have gained importance worldwide, not only because processing results in minimal CO_2 emissions [1], but also it improves durability and long-term strength [2]. The pozzolan activity of a natural mineral compound depends on its chemical composition, amorphous components, and fineness. Consequently, the mineralogy of pozzolan materials of geological origin is characterized by high content of amorphous silica, which, upon contact with water, reacts with calcium in a manner similar to Portland cement, forming hydrosilicates [2]. The high concentration of silica, alumina, and iron oxide in these hydrosilicates promotes pozzolan activity. A natural pozzolan is defined either as a raw or calcined material that has pozzolan properties, such as volcanic ash or pumicite, opaline chert, shales, tuffs, and some diatomaceous earths. According to the ASTM C618, the natural pozzolans in the raw or calcined state are designated as Class N pozzolans and are described in the specifications as: "Raw or calcined natural pozzolans that comply with the applicable requirements for the class as given herein, such as some diatomaceous earth; opaline chert and shales; tuffs and volcanic ashes or pumicites, any of which may or may not be processed by calcination; and various materials requiring calcination to induce satisfactory properties, such as some clays and shales [2,3]."

Figure 1 shows a summary of Uruguay's geology, showing a Precambrian basement divided into four Terranes: Piedra Alta, Cuchilla Dionisio, and Nico Pérez, separated by the Sarandí del Yí, Sierra Ballena and Colonia shear zones [4]. Folded sedimentary and/or very lowgrade metasedimentary deposits associated with the Ediacaran basins are located within the Nico Pérez and Cuchilla Dionisio Terranes (Table 1). These basins correlate with sedimentary

units of the Tandilia Terrane in Argentina and the Camaquã Basin in Brazil and comprising extended pelitic deposit of marine origin [5]. Paleozoic sedimentary deposits are characterized by extensive sequences of stratified clay-rich rocks, affected only by early diagenesis and without deformation. Notable examples include the Cordobés Formation (Devonian), composed of kaolinite, and the Yaguarí Formation (Permian), composed of mortmorillonite, which have been exploited for ceramics and bentonite production, respectively [6]. The Arequita Formation (Lower Cretaceous) composed of felsic lavas and pyroclastic rocks, is part of the Mesozoic magmatism widespread in the Paraná Basin and adjacent

areas, encompassing southern Brazil, Argentina, Paraguay, and Uruguay [4,7]. The original mineralogical composition of these units can undergo significant modification during hydrothermal alteration, metasomatism, diagenesis and/or metamorphism; therefore, conducting detailed mineralogical analyses is essential to verify material quality. In Uruguay, pozzolans have been commercialized as additives in the cement industry solely by the company Cementos Artigas SA. Precambrian pelites from the Don Mario Formation were exploited at the Cerro Verdún open-pit and subsequently activated by calcination [8], (Table 1).

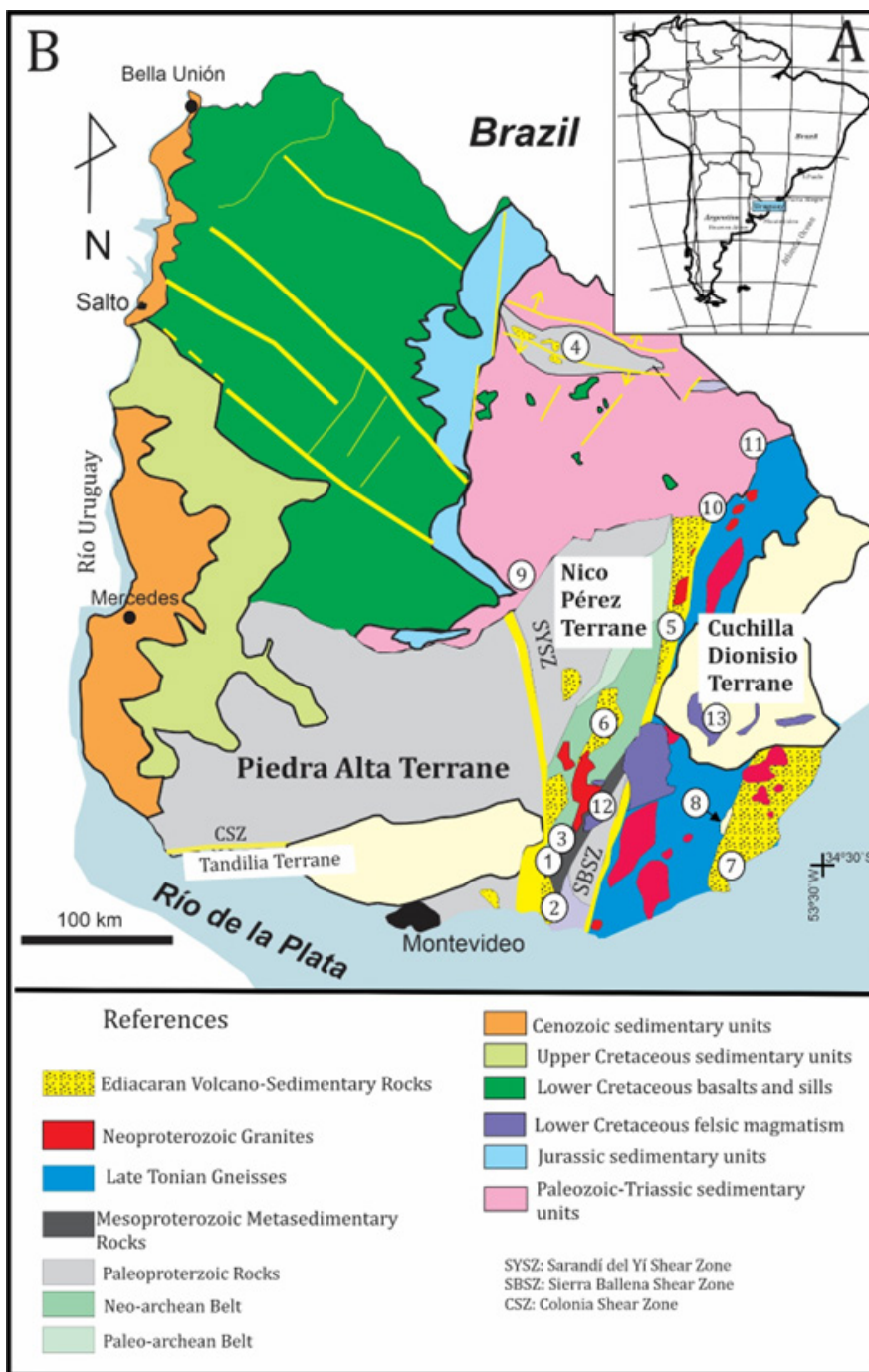


Figure 1: A. Location of Uruguay. B: Simplified Uruguayan geological map, modified from [4,5]. See Table 1 for location of the analyzed samples.

Table 1: Summary of geological units, location, grain size, mineralogical and major elements composition of the analyzed sample sin this work.

Geological Unit	Ref.	Lithology	Outcrop	Locality	Sample	Grain Size	Main. Min.	Acc. Min.	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	ΣSi+Al+Fe
Precambrian						µm			%	%	%	%	%	%	%	%
Don Mario Fm.	[7]	Pelites	1	Minas	PE44	40 ~70	qz, ill, cl, kFd	Py, mg, Ap	77.39	9.42	6.70	1.29	2.07	2.66	0.31	93.51
Las Ventanas Fm.	[11]	Pelites	2	C° Las Ventanas	LV1	35 ~60	qz, ill, cl, kFd, Pg	Py, Cc, Mc, Ba	64.29	13.27	6.63	7.57	2.80	3.36	1.80	84.19
Las Ventanas Fm.	[11]	Pelites	3	A° Campanero	LV2	20~45	qz, ill, cl, kFd, Pg	Py, ilm, Zr	71.78	13.98	6.62	0.32	1.39	3.00	2.98	92.38
Minas de Corrales Fm.	[9]	Pelites	4	Minas de Corrales	MC3	15~70	qz, ill, cl, kFd	hem, ilm, mg	77.05	14.42	2.07	0.15	0.71	4.65	0.27	93.54
Yerbal Fm.	[12]	Pelites	3	A° Campanero	YE1	30~90	qz, ill, cl, kFd	Mg, Zr	65.42	20.85	3.98	0.34	2.32	6.32	0.24	90.25
Yerbal Fm.	[12]	Pelites	5	Quebrada de los Cuervos	07_4	20~100	qz, ill, cl, kFd	Mg, Zr	81.03	12.44	2.24	0.33	0.72	2.04	1.11	95.71
Cerro Espuelitas Fm.	[12]	Pelites	6	Cerro Sepultura	17_2	10~35	qz, ill, cl, Bi, kFd	Mg, Zr	70.08	14.49	3.81	0.22	2.50	8.21	0.39	88.38
Cerro Espuelitas Fm.	[12]	Pelites	6	Cerro Sepultura	17_7	13~45	qz, ill, cl, kFd, Tr	Mg, Zr, Om	61.39	15.58	3.81	4.65	4.46	9.34	0.46	80.78
Rocha Fm.	[13]	Pelites	7	La Paloma	FR009	30~100	qz, Bi, cl, Pg	Ap, ilm, Ru	81.10	9.48	3.51	0.19	0.92	2.49	2.13	94.09
Cerros de Aguirre Fm.	[14]	Tuff	8	C°de Aguirre	CA1f	15~40	qz, kFd, ill, ms, sm	ilm, mg	74.79	14.71	5.96	0.08	0.45	3.45	0.55	95.46
Cerros de Aguirre Fm.	[14]	Tuff	8	C°de Aguirre	CA1g	50~180	qz, ill, ms, sm	ilm, mg	84.79	7.75	3.03	0.24	0.65	1.93	1.54	95.57
Paleozoic																
Cor-dobés Fm.	[8]	Claystone	9	Blanquillo	C1	1~10	Ka	ill, qz, kFd, Ru	52.26	25.92	1.99	0.2	0.96	3.53	0.14	80.17
San Gregorio Fm	[4]	Siltstone	10	Río Tacuarí	SG1	5~100	qz, Ka, ill	hem	87.48	10.10	1.12	0.03	0.13	0.64	0.09	98.70
Yaguarí Fm.	[8]	Claystone	11	Villa Viñoles	Y1	2~15	Mon	sm	60.21	18.63	4.12	3.29	4.63	0.22	0.11	82.96
Yaguarí Fm.	[8]	Claystone	11	Villa Viñoles	Y2	2~15	Mon	sm	60.39	18.57	3.59	3.15	5.36	0.00	0.1	82.55
Lower Cretaceous																
Arequita Fm.	[15]	Rhyolites	12	Marmaraja	Ar1	10~100	qz, san, Ka, ill, vg	Zr, ilm, kFd	77.51	12.03	2.08	0.14	0.18	4.94	3.34	91.62
Arequita Fm.	[15]	Rhyolites	13	Lascano	L1	50~150	qz, san, vg, ill-sm	Zr, ilm	80.60	9.03	2.62	0.35	0.24	6.47	0.69	92.25
Arequita Fm.	[15]	Rhyolites	13	Lascano	L2	50~500	qz, san, vg, ill-sm,	Zr, ilm	75.4	10.32	8.17	1.47	0.84	3.19	0.59	93.89

Scanning Electron Microscopy (SEM) investigations are based on the interaction of a focused electron beam under vacuum with a sample surface, leading to emission of Secondary Electrons (SE) and Back Scattered Electrons (BSE) from the surface. The Energy-Dispersive X-ray Spectrometry (EDS) attached to the SEM provides information on the topography, morphology, and chemical composition of both single grains and composite areas within the samples. In this work, representative rock samples from eleven geological units of Uruguay, encompassing Precambrian pelites and tuffs, Paleozoic claystones, and Lower Cretaceous felsic volcanic rocks, are compared using the SEM-EDS [9]. This approach enables a preliminary evaluation of the pozzolanic activity by: 1) the identification of the mineral phases and the amorphous material present, 2) the determination of grain size and its distribution, and 3) the quantification of the average major element's composition, specifically $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{FeO}$.

Materials and Methods

A total of 19 rock samples from 13 localities were collected (Figure 1 & Table 1) and processed at the Laboratorio de Geología del Departamento de Geociencias del Centro Universitario Regional del Este. To perform textural observations of the clay fraction, small rock fragments were mounted and gold sputtered. For elemental analysis, 30 μm thick, thin sections were prepared and polished using a diamond paste of 3 μm , 1 μm and 1/4 μm to obtain a mirror-

like surface; subsequently, these were carbon-coated for SEM observation. Back-Scattered Electron (BSE) imaging and elemental analysis of the polished thin sections and rock fragments were done using a Jeol NeoScope JCM-6000 Plus equipped with an Energy-Dispersive X-Ray Spectrometry (EDS) consisting of a 10mm² Silicon Drift Detector (SDD). Major element mapping and single-grain analysis of major and minor elements analysis were achieved according to established recommendations [10]. Electrons were emitted by a traditional tungsten filament with a working distance of 19mm. The X-ray take-off angle was approximately 25°. The energy resolution of the SDD was ~133eV at the energy of Mn K α (5894eV). The EDS analyses were conducted using a standard probe current (~30nA), a collection time of 60s, and an accelerating voltage of 15kV.

Results and Discussion

The comparative results of all samples are presented in Table 1. Precambrian pelites and tuffs, with a sum of $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ exceeding 70%, exhibit favorable geochemical conditions as precursors for natural pozzolans. The mineralogical composition shows that these units are primarily composed of illite, detrital quartz and potassium feldspar (Figure 2 & 3). The Paleozoic claystones of the Cordobés Formation are primarily composed of very fine-grained kaolinite, as evidenced by high Al_2O_3 content of 26% accompanied by minor amounts of illite (Figure 3E & 3F).

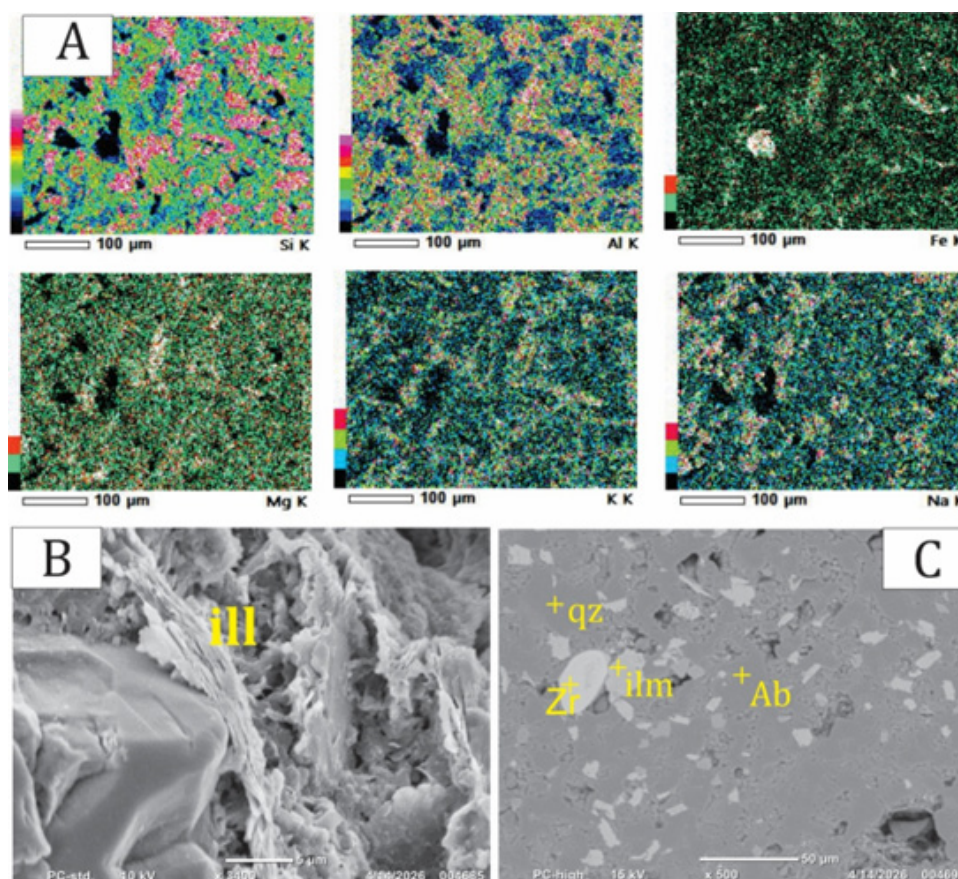


Figure 2: Metapelites of the Las Ventanas Formation (LV2). A: Elemental map showing the distribution of major elements. B: SEM image showing thin illite layers. C: EDS analysis on a polished thin section showing qz: quartz, Zr: zircon, ilm: ilmenite, Ab: albite.

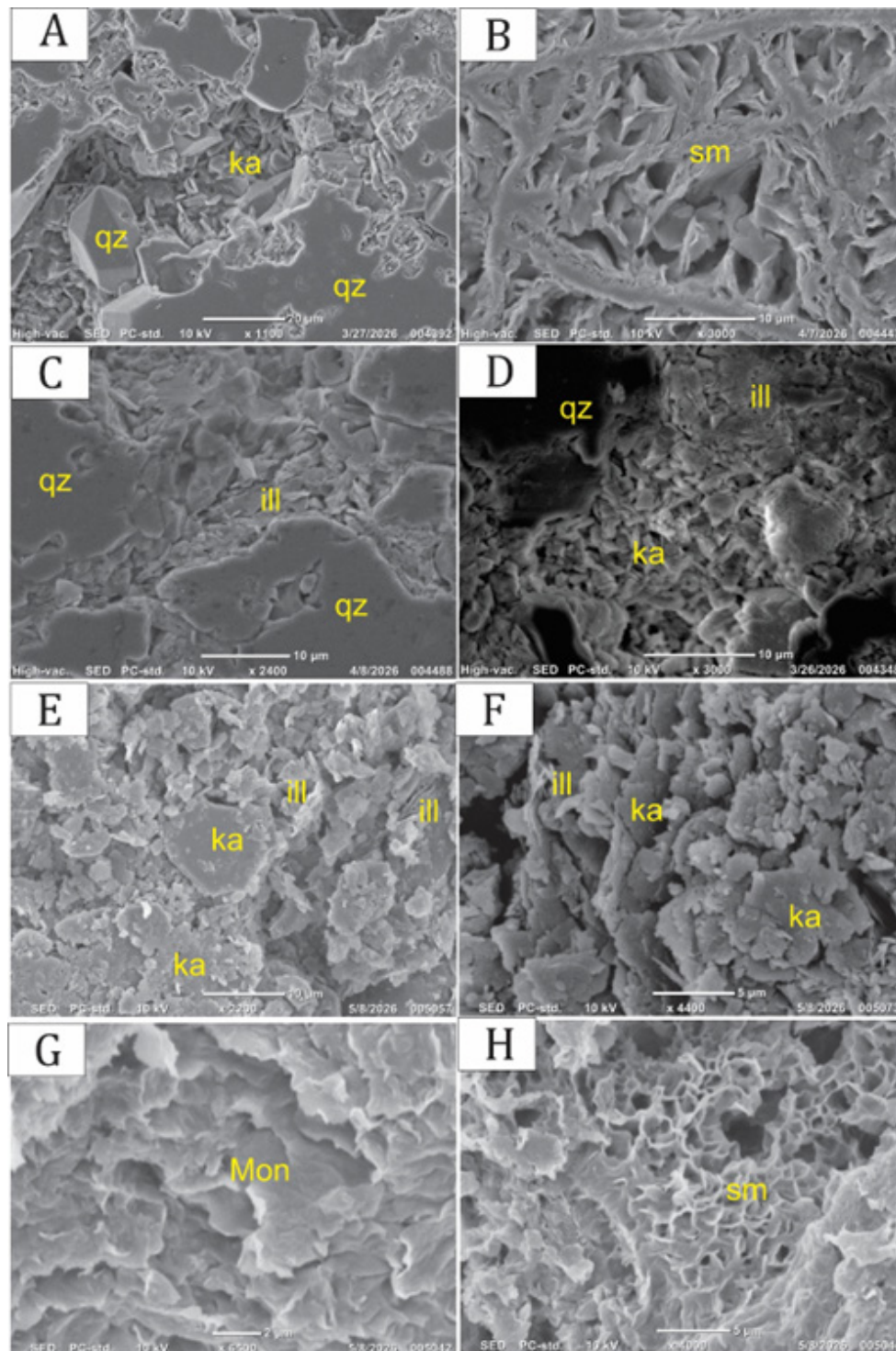


Figure 3: Clays identification using SEM images. A: and B Kaolinite (Ka), quartz (qz) and smectite (sm) of rhyolites from the Arequita Formation. C: illite (ill) and quartz from the Cerros de Aguirre Formation metatuff. D: Kaolinite of the San Gregorio Formation siltstones. E and F: Kaolinite crystal and Illite of Codobés Formation. G and H: Montmorillonite and honeycomb texture of smectite of Yaguari Formation.

Potassium feldspar altered to illite is also present. The siltstones of the San Gregorio Formation consist of an alternation of silt and clay layers (varves) of glacial origin. Very fine-grained detrital quartz is the main component, and potassium feldspar altered to kaolinite is very common. Both units exceed a total content of 70% for $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{FeO}$ oxides. The claystones of the Yaguari Formation are mainly composed of montmorillonite, followed in

order of abundance by smectites (Figure 3G & 3H); they are very fine-grained, yet their $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{FeO}$ content do not exceed 70%. The rhyolites of the Arequita Formation are mainly composed of quartz and sanidine immersed in a glassy matrix; this matrix is mostly altered to kaolinite at the Marmarajá site and to smectites at the Lascano site (Figure 3A & 3B). All three samples exhibit a $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ oxide content well above 70%.

Mineralogical and geochemical results for the Precambrian rocks show a consistent composition across the various units analyzed. The samples here analyzed exceed the 70% minimum requirements for the sum of SiO_2 , Al_2O_3 , and Fe_2O_3 oxides of [3]. Furthermore, they show similar values when compared to whole-rock geochemistry data from the literature (Table 2). Given their vast areal extent and distribution, this work suggests a high prospecting potential for natural pozzolanic materials within the Ediacaran metapelites of the Nico Pérez Terrane. A similar mineralogical composition, dominated by illite and fine-grained

quartz and feldspar, was observed for the Minas de Corrales, Las Ventanas, Yerbal and Cerro Espuelitas Formations. It is worth noting that illite-dominated pelites (shales) can exhibit pozzolanic activity after thermal treatment at 950 °C, as seen in samples of the Cerro Largo and Cerro Negro Formations in Buenos Aires, Argentina [11]. These Ediacaran units are correlative in age with the Yerbal, Cerro Espuelitas, Las Ventanas and Minas de Corrales Formations; they were likely deposited in a similar shallow marine environment and exhibit almost identical mineralogical characteristics [5,12].

Table 2: Representative XRF analysis take from previous works.

Geological Unit	Ref.	Lithology	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	$\Sigma\text{Si}+\text{Al}+\text{Fe}$
			%	%	%	%	%	%	%	%
Don Mario Fm.	[7]	Pelites	62.43	14.29	4.63	1.19	2.86	2.84	1.1	81.35
Las Ventanas Fm.	[11]	Pelites	61.60	14.60	5.13	1.72	1.547	3.34	2.34	81.32
Minas de Corrales Fm.	[11]	Pelites	65.48	15.79	7.04	0.04	0.83	4.91	0.26	88.31
Yerbal Fm.	[12]	Pelites	67.46	17.02	5.84	0.27	0.59	2.31	2.87	90.32
Cerro Espuelitas Fm.	[12]	Pelites	62.60	17.09	3.51	0.09	2.38	9.18	2.23	83.20
Rocha Fm.	[13]	Pelites	70.10	12.69	4.44	0.42	1.68	2.68	3.00	87.23
Cerros de Aguirre Fm.	[14]	Pelites	74.06	15.98	1.19	n.d.	0.53	3.89	0.14	91.23
Cordobés Fm.	[8]	Claystones	57.00	25.50	1.50	n.d.	n.d.	3.1	n.d.	84.00
Yaguarí Fm.	[21]	Claystones	59.64	17.75	3.94	2.46	6.54	0.11	n.d.	81.33
Arequita Fm.	[17]	Rhyolites	77.16	12.06	2.05	0.11	0.07	4.92	3.48	91.27

Due to its $\text{Al}_2\text{O}_3:2\text{SiO}_2$ ratio and high kaolinite content (>90%), the El Cordobés Formation claystones could be used for the manufacture of metakaolin. When heated to temperatures between 600 and 900 °C, this material can generate a highly reactive pozzolanic product [13]. The Yaguarí Formation is primarily composed of montmorillonite; however, its use as a natural pozzolan could be problematic, as untreated clays have been unsatisfactory in terms of reactivity and workability. Nevertheless, the material could be transformed through calcination, following a typical thermal transformation sequence of dehydration, dehydroxylation, amorphization, and subsequent crystallization [14].

Among the geological materials that can be used as natural pozzolans in their raw state, those of felsic volcanic origin, such as rhyolites, are particularly useful due to the high reactivity of the volcanic glass when its concentration exceeds 40% of the total weight. The most recent geological record of acidic volcanism in Uruguay occurred during the Lower Cretaceous, recorded by the Arequita Formation. This unit is mainly composed of quartz, sanidine, and altered glassy material, and underwent transformations during diagenesis or weathering. The precise identification of alteration products and volcanic glass via SEM-EDS indicates a high potential for prospecting these volcanic rocks. Moreover, rhyolitic rocks with a glass content between 40 and 100 % require no calcination for pozzolanic activation [1].

Relict glassy material was detected in this study, although mostly devitrified and transformed into clays (illite-smectite, Figure 4). Moreover, previous work on unit's correlative to the

Arequita Formation in Brazil, such as the Serra Geral Formation have shown its potential use as pozzolan after thermal treatment [15]. The quantification of volcanic glass in the Arequita Formation serves as an exploration guide for natural pozzolanic materials, as the preservation and distribution of the volcanic glass is not homogenous across the different outcrop sites.

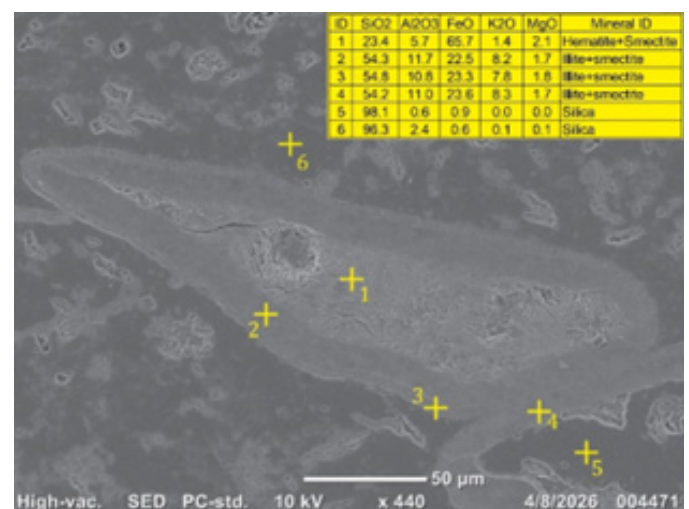


Figure 4: SEM image and EDS analyses of the Arequita Formation rhyolites showing ignimbrite texture, evidence of devitrification (1) and illite-smectite formation (2, 3 and 4), within a matrix of poorly crystalline silica paste (5 and 6) at the Lascano locality.

Conclusion

An initial assessment of several geological materials of Uruguay using SEM-EDS provides critical information regarding: 1) the identification and composition of mineral phases, including alteration products and accessory minerals; 2) the presence of vitreous and/or amorphous material; 3) the compositional and textural characterization of clays (e.g., kaolinite, smectite, illite); 4) the estimation of the average composition and distribution of the major oxides ($\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$) in the sample [16,17]. The methodology applied serves as a preliminary exploration guide for geological materials in an area of interest, leading to a rational use of pozzolanic index tests on selected samples (e.g. 28-day strength activity index with Portland cement [1]), which are otherwise expensive and time-consuming [18-21].

The sample set analyzed in this work identifies specific targets for the exploration of sedimentary and volcanic rocks that could be processed to produce natural pozzolan in Uruguay:

- Precambrian metapelites: fine-grained and mostly composed of illite, feldspar, and quartz; require a thermal treatment ($\sim 950^\circ\text{C}$) to produce pozzolanic geomaterial.
- Claystones of the Cordobés Formation: composed of 90% kaolinite and 10% illite, emerge as an ideal candidate as the transformation to metakaolin (a highly reactive pozzolan) requires calcination at temperatures close to 600°C .
- Rhyolites of the Arequita Formation: the glass content is a critical parameter in the exploration of this unit. Samples with over 40% glass may require minimal or no thermal treatment to achieve adequate pozzolanic reactivity.

Acknowledgement

The authors thank the Universidad de la República and PEDECIBA Geociencias for their support of this work. The reviewers and the Editor are deeply acknowledged.

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